AD-A043 244 GENERAL ELECTRIC CORPORATE RESEARCH AND DEVELOPMENT --ETC F/G 20/2
IMPROVEMENT OF GAAS CRYSTAL QUALITY BY MEANS OF LIQUID-SOLID IN--ETC(U)
JUL 77 W TANTRAPORN F44620-76-C-0127

UNCLASSIFIED SRD-77-116 AFOSR-TR-77-0969 NL

| OF |
ADA
043244

END
DATE
FILMED
9-77
DDC

AFOSR-TR- 77-0969

IMPROVEMENT OF GaAS CRYSTAL QUALITY BY MEANS OF LIQUID-SOLID INTERFACE CURVATURE CONTROL

FINAL REPORT

This document is an interim summary of work which is being continued with AFOSR funding under Contract No. F49620-77-C-0105

Prepared by

W. Tantraporn

Period Covered

June 1, 1976 through May 31, 1977

DEPARTMENT OF THE AIR FORCE
AIR FORCE OFFICE OF SCIENTIFIC RESEARCH
BOLLING AIR FORCE BASE, D.C. 20332

Contract No. F44620-76-C-0127

General Electric Company
Corporate Research and Development
Schenectady, NY 12301

SRD-77-116

July 1977

AIR FORCE OFFICE OF SCIENTIFIC RESEARCH (AFSC)
NOTICE OF TRANSMITTAL TO DDC
This technical report has been reviewed and is
approved for public release IAW AFR 190-12 (7b).
Distribution is unlimited.
A. D. BLOSE
Technical Information Officer

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER AFOSR-TR- 77-0969	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) IMPROVEMENT OF GaAS CRYSTAL QUALITY BY MEANS OF LIQUID-SOLID INTERFACE CURVATURE CONTROL.		5. TYPE OF REPORT & PERIOD COVERED FINAL
		6. PERFORMING ORG. REPORT NUMBER SRD-77-116
7. AUTHOR(s) Tantraporn, W.		8. CONTRACT OR GRANT NUMBER(s) ~~F44620-76-C-0127~~ new F49620-77-C-0105
9. PERFORMING ORGANIZATION NAME AND ADDRESS General Electric ~~Company~~ Corporate Research And Development, PO BOX 8 Schenectady, NY 12301		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 61102F-2306-B1
11. CONTROLLING OFFICE NAME AND ADDRESS AFOSR/NE Bolling AFB, DC 20332		12. REPORT DATE JULY 77
		13. NUMBER OF PAGES 7
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) Final rept. 1 Jun 76-31 May 77		15. SECURITY CLASS. (of this report) UNCLAS 12 11 p.
		15a. DECLASSIFICATION DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

16 2306 17 B1

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Crystal Growth
Gallium Arsenide

406617

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A solid-convex condition for growth of GaAs single crystal from the melt is sought, so that crystal quality may improve as a function of length. A short-dipole-like thermal configuration with calibrated temperature profile was used in the vertical zone melting technique, but was abandoned because equilibrium vapor transport could not be maintained. Solidification of long melt from one end across the melting point equitemperature surface also failed to yeild good crystals. The melting point equitemperature surface's curvature cannot be

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

20. maintained as the medium changes from air to GaAs, i.e., when the sample tip enters the surface.

The second year effort includes two means to produce and hold the melting point equitemperature surface of appropriate curvature.

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

Section 1

ABSTRACT

The objective of this program is to study the effect of liquid-solid interface curvature on crystal quality. In particular, it is to be determined whether a solid-convex curvature growth condition yields a defect density that is a decreasing function of the length of the GaAs crystal grown.

A vertical zone melting technique was adopted. The furnace was designed to provide a short-dipole-like heat flow that yields curved equitemperature contours. The growth rate was controlled with a specially designed vertical furnace movement that minimizes sample vibrations.

The results indicate that the temperature profile required for a solid-convex growth condition is difficult to achieve due to thermal conductivity inhomogeneities associated with different positions of the GaAs sample. The cause of the resulting adverse (solid-concave) contour was shown to be the region of inhomogeneous thermal conductivity in the vicinity of the melting-point equitemperature contour during passage of the sample tip through the contour. Two means to alleviate the problem have been proposed and constitute the bulk of a second year's effort.

Section 2

OBJECTIVES AND ACCOMPLISHMENTS

The general objective of this program is to study the effect of liquid-solid interface curvature on GaAs crystal quality. It is believed that a solid-convex curvature growth condition will yield high quality, single-crystal GaAs; the defect density should be a decreasing function of the length of the grown crystal.

A short-dipole-like heat flow condition, as shown in Figure 1, provides temperature contours of various types; a desirable contour curvature can be selected for study by simply adjusting the furnace power such that the desired contour is at the melting temperature.

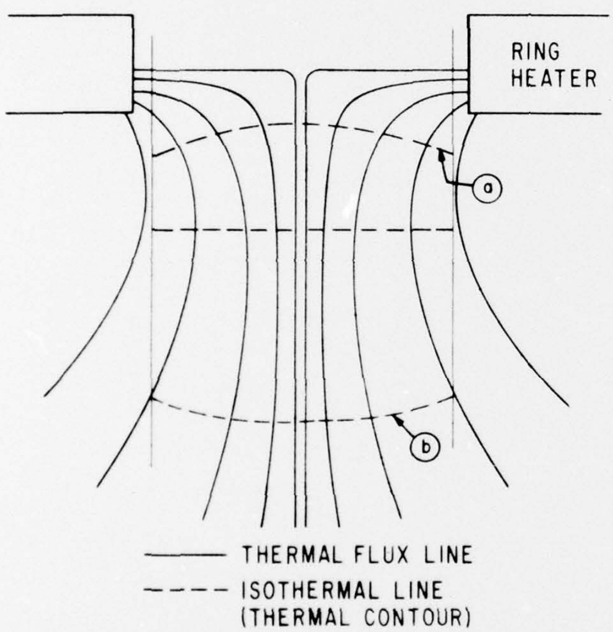

Figure 1.

"Short-dipole" Thermal Configuration. If melting point is at isothermal line (a), interface is solid-convex, at (b) solid-concave

The proposed experimental program consisted of the following steps:

1. Design and construction of a vertical movement to support the existing, calibrated furnace so that the growth rate can be selected and controlled.
2. Design and construction of a sample holder to monitor and minimize vibrations.
3. Recalibration of the temperature profile (cylindrical coordinates r, z).
4. Regrowth runs using polycrystalline GaAs in a sealed evacuated quartz tube tapered to a point at the lower end (to provide an arbitrarily oriented seed) under various temperature and growth speed conditions.
5. Evaluation of the crystal quality by x-rays and etch-pit density counting.
6. Establishment of the best growth conditions and production of high-quality GaAs crystals.

Step 1 of the experimental program was successfully completed. The calibrated movement provides a constant speed of from 0.35 mm/h to 32 mm/h over a vertical distance of 12 inches with vertical "play" limited to less than 20 mils (Figure 2).

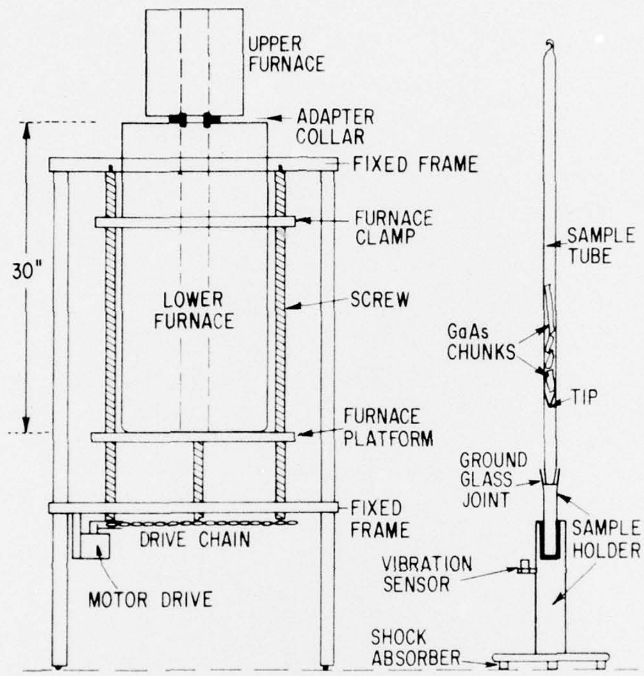

Figure 2. Schematic Diagram of Crystal Growth Apparatus and Sample Holder

Step 2 was also successfully completed. A series of experiments resulted in the choice of a simple air-cushion, three-leg, shock mounting. A vibration monitor capable of recording the logarithmic amplitude of sample vibrations at frequencies from 0 to 100 kHz was built. The mechanical vibrations of the sample holder were reduced by shock mounting to a level limited by noises in the air.

In Step 3, experiments were performed and the temperature profile of the furnace was established.

Step 4 consisted of a number of experiments. Crystal regrowth was carried out twice according to the originally proposed scheme. Poor results were obtained from the first run due to a less than optimal cold-zone temperature used to regulate the As pressure. The second run resulted in an explosion during the hot-zone's downward movement. The short liquid zone was followed by a solidfying GaAs plug that closed off the equilibrium transport of As to the cold zone and resulted in a high-pressure build-up in the melt.

The furnace vertical temperature profile was then modified to insure that the GaAs initially be liquid over the entire sample length. The temperature contour curvature at the lower end of the furnace was found to be less than that at the corresponding position with the short-dipole-like configuration, but still appreciable.

The third and fourth regrowth runs were made with the new furnace profile. Vertical variations existed in the resulting ingot, which were visible as bands of various shades of silver and grey (Figure 3). The silvery coating starting at the tip was identified as excess As precipitating out during crystallization. This indicated that the temperature of the melt was not constant despite temperature control to better than ± 0.5 C at the thermocouple position.

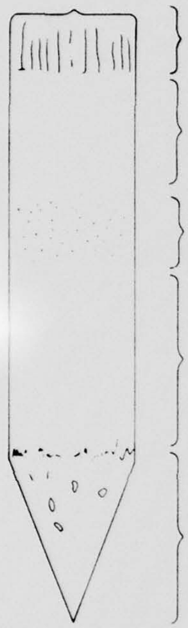

Figure 3.

Sketch of GaAs Ingot Obtained from Run 3 (total length 7 inches) Showing Bands of Different Surface Morphologies

The thermocouple had been located at a position where the melting temperature contour was expected, thus control could be exerted at the crystallizing interface. However, this position allows the melt's temperature to have a wide range, depending on the temperature gradients across the melting-point equitemperature surface. These temperature gradients, in turn, depend on thermal conductivity nonuniformities. During the period before, during, and after the sample tip is in the controlled zone, the controlled zone is comprised of air, air plus the sample cone, and the sample rod, respectively. Thus, the temperature above the controlled zone can vary widely during the growth procedure.

An additional control zone has now been incorporated in the new furnace. Other refinements also have been incorporated. For example, it was found that seasonal temperature variations also affect the furnace temperature with a positive feedback enhancing the variation. The room temperature is now being carefully regulated.

The thermal conductivity nonuniformities not only affect the z-direction thermal profile discussed above but also produce adverse profile curvature. This is illustrated in Figure 4. Here a uniform one-dimensional heat flow is shown perturbed by the presence of the higher conductivity sample tip. The flat temperature profile in air at the interface temperature, T_i, becomes solid-concave in the conical section of the sample. Because the thermal conductivity disparity between GaAs and air is appreciable, the solid-convex type of temperature contour originally present in the air-core furnace can be overcome by the presence of the sample cone, and a solid-concave-type contour

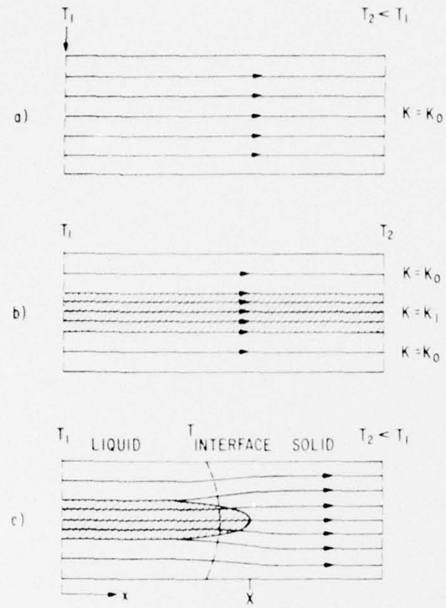

Figure 4

Effect of Sample Tip on Isothermal Profile. In a) and b), isothermal profile is flat and normal to the heat flow. In c), sample tip would cause the profile to be solid-concave

results. It is believed that this occurs in the present growth system and prevents us from producing the desired solid-convex profile.

Corrective action involving 1) a secondary seeding step after the shoulder portion of the sample has passed through the T_i contour (Figure 5) and 2) the addition of a supplementary material below (but in intimate contact with) the conical section in order to minimize thermal conductivity disparities constitute the bulk of the work for the second year. A new technique for determining the temperature contour in the solid rod will also be incorporated. It is believed that the solid-convex type of contour can be established and the original study objective fulfilled.

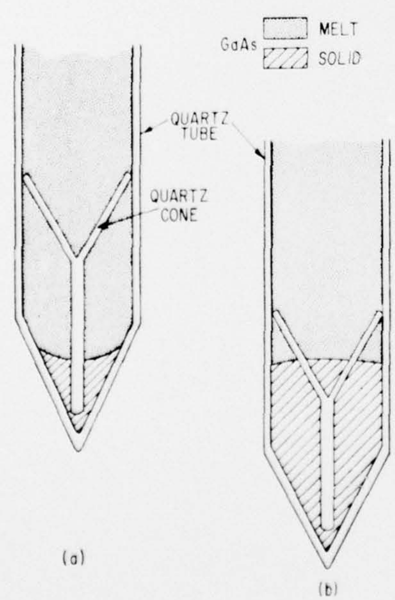

Figure 5

Proposed Secondary Seeding Technique. In (a) interface isotherm is solid-concave (cf Figure 4c), while in (b) isotherm is far from sample tip and can assume curvature of solid-convex isotherm resulting from furnace winding

Step 5 of the originally proposed experimental program was carried out to the limited extent required with the samples available. X-ray diffraction measurements were made to check the size of the crystals in the polycrystalline regrowth ingot of run 3 and showed that the ingot consists of a few large single crystals. The polycrystallinity of the regrown ingot is attributed to multiple seedings caused by the improper T_i contour. Etch-pit counting has not been carried out; the crystals obtained so far have been obviously poor. Auger electron analysis was performed to identify the surface layer's composition in the various "bands" mentioned earlier.

At the end of the first year, the furnace was rewound with new all Pt windings. They replaced the burned-out section of the furnace originally designed to be the lower temperature portion in the short-dipole-like heat-flow configuration but later used at higher temperatures. A rig was designed and constructed to measure the temperature profile curvature in solids. Another rig was designed and constructed to measure the thermal conductivity of various composites that are potential supplementary materials for use in minimizing the thermal conductivity nonuniformities. Thus, we are ready to carry out the second year's program.

Section 3

PERSONNEL

PROJECT PERSONNEL

W. Tantraporn
W.R. Cady
J. Bortscheller

INTERACTIONS WITH DOD PERSONNEL

U.S. Air Force Avionics Laboratory
Elizabeth H. Tarrants